PERFORMANCE CHARACTERIZATION METHODS OF AEROSOL SAMPLERS

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ABSTRACT

Samplers are characterized at ECBC using the following methods: (1) monodisperse fluorescent/non-fluorescent PSL microspheres with fluorometric analysis or Counter analysis, (2) polydisperse solid aluminum oxide particles with APS analysis or Coulter Multisizer analysis, (3) fluorescent oleic acid particles with fluorometric analysis, and (4) bioparticles with Coulter Multisizer, culturing, ELISA, PCR, and APS analyses. PSL microspheres are generated using the Collison nebulizer, sonic nozzle, Ink Jet Aerosol Generator (IJAG), and puffers. Aluminum oxide particles are generated using the sonic nozzle, fluorescent oleic acid particles are generated using the Vibrating Orifice Aerosol Generator, and bioparticles are generated using the puffers, IJAG, and sonic nozzle.

INTRODUCTION

Air samplers are gaining importance in the war against terrorism and on the battlefields to detect the presence of chemical, biological, and nuclear aerosols. Samplers and detection systems must be tested and their performance efficiencies determined so that suitable samplers and detectors can be used at each condition. Knowledge and use of efficient air samplers enhance the ability to protect soldiers, first responders, and the general public.

The performance of an aerosol sampler is the product of the sampler's aspiration, transmission, and collection efficiencies. The aspiration efficiency of a sampler gives the efficiency with which particles enter into the sampler inlet; transmission efficiency gives the efficiency with which the particles are transported to the collection point, and the collection efficiency gives the efficiency with which particles are captured and retained by the sampling medium.

The samplers are characterized in wind tunnels to determine the effect of wind speeds on aspiration efficiencies of the sampler inlets and in chambers to determine the sampling efficiency at calm air conditions. Tests are also conducted in flow-through cells to determine sampler component efficiencies. This paper describes the methods used at the Edgewood Chemical Biological Center (ECBC) chambers to characterize aerosol samplers at calm air conditions.

METHODS

Sampling efficiency tests at calm air conditions are conducted at ECBC in a 70 m^3 chamber using many methods. The methods used at ECBC are: (1) monodisperse fluorescent and non-fluorescent polystyrene latex (PSL) microspheres with fluorometric analysis or Coulter anaysis, (2) polydisperse solid

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aluminum oxide particles with Aerodynamic Particle Sizer (APS) or Coulter Multisizer analysis, (3) fluorescent oleic acid particles with fluorometric analysis, (4) bioparticles with culturing, ELISA, PCR, Coulter Multisizer and APS analyses. APS analysis is only used in cases where the sampler is an aerosol concentrator with the concentrated aerosol output.

The aerosols are generated using many methods. Monodisperse PSL microspheres are generated using a 24 or 36 jet Collison nebulizer, sonic nozzle, Ink Jet Aerosol Generator (IJAG), and puffers. Polydisperse solid aluminum oxide particles are generated using a sonic nozzle, fluorescent oleic acid particles are generated using a Vibrating Orifice Aerosol Generator (VOAG), and bioparticles are generated using the puffers, IJAG, and sonic nozzle.

The sampling efficiency tests are conducted by generating the aerosol for a certain time, mixing the aerosol in the chamber for one minute to achieve uniform aerosol concentration in the chamber, and then sampling from the chamber using the samplers and the reference filters. The collected samples are analyzed and sampling efficiency is determined by comparing the particles collected by the sampler to particles collected by the reference filters. The airflow rate of reference filters, the airflow rate of samplers, the liquid volumes of samples and the reference filters are taken into account in the sampling efficiency calculations.

To achieve accurate sampling efficiency results, the airflow rates must also be measured carefully. The airflow rates of the reference filters and samplers are measured using a Buck calibrator (A.P. Buck, Inc., Orlando, FL) and a Kurz air flow meter (Kurz Instruments, Inc., Monterey, CA). Other characteristics of the samplers are also measured. The weight and dimensions of the samplers are measured and the power usages are measured using a power meter (Extech Instruments, Taiwan).

CHAMBERS AND EQUIPMENT

Chambers and equipment used in characterizing samplers at ECBC are described in this section. A 70 m^3 chamber is used in most of the sampler characterization tests. There are also smaller stainless steel and plexiglass chambers available if very high concentration aerosols are needed for low air flow rate sampler tests.

70 m^3 Chamber: Sampler characterization tests were conducted in a 70 m^3 Bio-Safety Level 1 (BL 1) chamber. Temperature and humidity of the chamber can be set and maintained easily and accurately by a computer. This computer also controls power receptacles inside the chamber.

HEPA filters are installed at the inlet to filter air entering the chamber to achieve very low background particle concentrations in the chamber. Similarly, HEPA filters are installed at the exhaust port to filter all particles leaving the chamber. The chamber aerosol is cleaned by exhausting the chamber air through the HEPA filters, and by pumping HEPA filtered air into the chamber. The maximum amount of airflow that can be exhausted from the chamber is approximately $2x10^4$ L/min by the exhaust pump. There is also a small re-circulation system that removes air from the chamber, passes it through a HEPA filter, and delivers it back to the chamber. This system is useful when the aerosol concentration in the chamber needs to be reduced incrementally.

Aerosols can be generated outside and delivered to the chamber, or can be generated inside the chamber. Fans mix the chamber air after and/or during the aerosol generation to achieve uniform aerosol concentration in the chamber. Previous tests showed that mixing the aerosol in the chamber for 1 minute is adequate to achieve uniform aerosol concentration.

Nebulizers: A 36 or 24 jet Collison nebulizer (BGI Inc, Waltham, MA) is used at ECBC to generate the PSL aerosols. PSL microspheres are added to de-ionized water and are used in the nebulizer. The Collison nebulizer is connected to compressed air and the compressed air exits from small holes inside the nebulizer at high velocity. The low pressure created in the exit region by the Bernoulli effect causes liquid to be drawn from the bottom of the nebulizer through a second tube. The liquid exits the tube as a thin filament that is stretched out as it is accelerated in the air stream until it breaks into droplets. The spray stream is directed onto the wall where larger droplets are impacted and removed from the air. The PSL microspheres are carried out of the nebulizer. Particles generated by this method are charged, so passing them through a Kr-85 radioactive neutralizer neutralizes them.

Sonic Nozzles: Dry aluminum oxide, PSL microsphers, and bio particles are aerosolized using a two fluid pneumatic sonic nozzle (SRI International, Menlo Park, California). It is connected to compressed air and the air exits out through a small annular opening. The low pressure created in the exit region by the Bernoulli effect causes powder to be pulled through an axial tube at a very low feed rate. Desired air to powder mass ratio is 80-100 : 1. Because the air flow rate (1100 L/min) and the aerosol generation rate are high, particles generated by this method are highly charged and they cannot be neutralized using the Kr-85 neutralizer.

Puffers: Puffers are metered dose inhaler (MDI) adapted by ECBC for use with biological simulants. The puffer is a portable convenient aerosol source and ejects a small reproducible cloud of simulant aerosol particles at the push of a button. A puffer releases a 60 mg spray of its contents into the air when it is activated. The droplets expand and evaporate instantly, leaving a small cloud of the loaded particles. It can be used as a quick source of particles in the laboratory and confidence checker in equipment deployed in the field. A puffer could also be used to fill a small chamber for more quantitative experiments. We have used many materials in the puffers such as Bg (Bacillus subtilis var niger), ovalbumine, and PSL microspheres. A typical formulation is 0.1% by weight of simulant material in 1,1,1,2-tetrafluoroethane (HFA-134a) as the propellant. The shot-to-shot mass variation is small, about 10%.

Ink Jet Aerosol Generators (IJAG): The Ink Jet Aerosol Generator was developed at ECBC for low concentration aerosol applications, however, it can also be used in some mid and high concentration applications. In aerosol concentrator characterization tests, the IJAG is typically used to generate particles directly into the aerosol concentrator.

A 12-nozzle ink jet cartridge (HP 51612A), purchased empty, is filled with a slurry of clean water and the material of interest. Droplets are fired downward through a heated drying tube where the water evaporates leaving an aggregate residue particle. Since the size of the primary ink jet droplet is fixed, about 50 micrometer diameter, the size of the residue particle depends only on the concentration of the slurry. We have prepared different concentrations of slurries to produce different size monodisperse particles. IJAG is capable of producing monodisperse particles at a range of 1 to 2,000 particles/sec for our tests.

Vibrating Orifice Aerosol Generator: A model 3450 Vibrating Orifice Aerosol Generator (VOAG) (TSI Incorporated, St. Paul, MN) is used in generating monodisperse fluorescent oleic acid droplets. The generated droplets pass through a Kr-85 neutralizer before entering the chamber to neutralize the charged particles.

Different concentrations of sodium fluorescein, oleic acid, and propanol are used to generate different size particles. The solution to be aerosolized is placed in a pressure container. The pressure forces the solution at a constant flow rate from the pressure container through a membrane filter and Teflon tubing into the liquid orifice assembly. The solution is forced through a small orifice to form a

liquid jet. A piezoelectric crystal produces mechanical vibration in the liquid orifice disk. The vibration causes the liquid jet to break up into uniform droplets. The alcohol evaporates leaving the fluorescent-tagged oleic acid as the final challenge particle. Changing the concentration of oleic acid in propanol, the vibration frequency of the liquid orifice disk, and liquid feed rate can change the particle size of the aerosols.

Fluorometer: The Turner Model 450 Fluorometer (Barnstead/Thermolyne Corporation, Dubuque, IA) is used to measure the fluorescence of the collected samples and reference filters. Appropriate excitation and emission filters are used to detect the blue (Ex:NB360; Em:SC430) and green (Ex:NB460; Em: SC500) PSL microspheres and sodium fluorescein (Ex:NB490; Em:SC515). When measuring sodium fluorescein, the pH of the sample must be between 8-10 to achieve maximum fluorescence. The temperature also affects the amount of fluorescence, therefore, the samples that are compared should be at the same temperature.

Coulter Multisizer: The Coulter Multisizer® II analyzer is a multichannel particle size analyzer (Beckman Coulter, Miami, FL) that uses electrical impedance as a method of measurement to provide a particle size distribution analysis. There are different size orifice tubes to measure different range of particle sizes. We generally use the 50-micron aperture diameter orifice tube in the Coulter Multisizer that measures 1 to 30 micron diameter particles.

The Coulter Multisizer analysis method requires that the sample be in electrolyte solution for analysis. This is achieved by either using the electrolyte solution as the sample collection liquid in the sampler or by diluting high concentrations of samples in electrolyte solution. Filters are processed in the electrolyte solution to remove the particles from the filter into the solution for Coulter Multisizer analysis. The measured geometric diameter is converted to aerodynamic diameter using the density of the particles and the shape factor of the particles. The disassociation of agglomerated and clustered particles in the electrolytic would not be desired and should be checked before using the Coulter Multisizer as the analysis method.

APS: The APS Model 3320 (TSI Incorporated, St. Paul, MN) is a high performance general-purpose particle spectrometer that measures both aerodynamic diameter and light-scattering intensity. The APS provides accurate particle count and size distributions for particles with aerodynamic diameters from 0.5 to 20 micrometer. It detects light-scattering intensity for particles from 0.3 to 20 microns. APS is specifically engineered to perform aerodynamic size measurements in real time using low particle accelerations.

SAMPLER EFFICIENCY TEST METHODS

Polystyrene Latex Microsphere (PSL) Tests**:** Sampling efficiency tests are conducted with monodisperse fluorescent and non-fluorescent PSL microspheres (Duke Scientific Corp., Palo Alto, CA). The PSL aerosols are generated using a 24 or 36 jet Collison nebulizer, sonic nozzle, IJAG, and puffers. The PSL microspheres have to be in powder form for the use of the sonic nozzle in aerosol generation. In general, we use the 36 jet collision nebulizer for generating the aerosols because the low air flow rate allows the aerosol to be neutralized using a Kr-85 neutralizer before it enters the chamber. During the experiments, aerosols were generated for 10 to 20 min, and the chamber air was mixed for 1 minute to achieve uniform aerosol concentration before sampling using reference filters and those samplers being tested.

Polycarbonate membrane filters (Osmonics Inc., Minnetonka, Minnesota) are used as reference filters to collect the fluorescent PSL microspheres if the analysis is by fluorometry or by Coulter Multisizer. After sampling, the sample liquid and reference filters are collected. The fluorometer or Coulter Multisizer directly analyzes sample liquids, however the membrane filters are processed to

remove microspheres from the filters into the liquid for fluorometer or coulter analysis[1]. The removal procedure consists of placing the membrane filters into 15 mL of filtered deionized water or electrolyte solution, then hand shaking for 10 sec followed by vortexing for 50 sec. The 60 sec of hand shaking and vortexing are repeated four times (total of 5 minutes) to completely remove fluorescent PSL microspheres from the membrane filters.

Polydisperse solid aerosols tests: Polydisperse aluminum oxide (Saint-Gobain Industrial Ceramics, Worcester, MA) aerosol is generated using the sonic nozzle into the 70 m^3 chamber. One gram of powder takes approximately 10 – 15 sec to be aerosolized. The generated aerosol is mixed in the chamber for one minute before sampling. Polycarbonate membrane filters (Osmonics Inc., Minnetonka, Minnesota) are used as reference filters as described above. Collected samples are analyzed using the Coulter Multisizer. The measured geometric diameter of aluminum oxide by the Coulter Multisizer is converted to aerodynamic diameter using the density of aluminum oxide (4 g/cm^3) and the shape factor (1.22). Currently the aerosols are not neutralized, however, we are in the process of designing and building a neutralizer for the sonic nozzle.

Sodium Fluorescein Tagged Oleic Acid (Fluorescent Oleic Acid) Tests: The monodisperse fluorescent oleic acid particles are generated using a Vibrating Orifice Aerosol Generator (VOAG, TSI Inc., St. Paul, MN). VOAG can be used to generate monodisperse 3 to >20 micron diameter particles. Sizes of the fluorescent oleic acid particles are determined by sampling the aerosol onto a microscope slide inserted into an impactor, and measuring the droplet size using a microscope. The measured fluorescent oleic acid particle diameter is converted to an aerodynamic particle size using a spread factor[3] and the density of fluorescent oleic acid. At the end of aerosol generation, the aerosol in the chamber is mixed for one minute to achieve uniform aerosol concentration. The samplers and the corresponding reference filters sample the aerosol simultaneously and for the same amount of time.

Glass fiber filters (Pall Corporation, Ann Arbor, MI) were used as the reference filters to collect the fluorescent oleic acid particles. After sampling, the filters were removed from filter holders, placed into a fluorescein recovery solution, and shaken on a table rotator (Lab-Line Instruments, Inc., Melrose Park, IL) for one hour. The recovery solution used in the tests also contained water with a solution pH between 8 and 10, obtained by adding a small amount of NH_4OH (e.g., 1000 mL of water with 0.563 mL of 14.8 N NH_4OH). Factors that affect fluorescein analysis and the removal of fluorescein from filters are described in detail by Kesavan et al. (2001)[2]. The samples from the aerosol samplers were pH corrected by adding NH_4OH before the amount of fluorescence was measured by the fluorometer (Barnstead/ Thermolyne, Dubuque, IA). All the samples were analyzed the same day as the experiment or the next day. In appropriate situations, the recovery solution is used as the sample collection liquid. Separate tests did not show any photo degradation of fluorescence with time under normal laboratory lighting.

Bioaerosol Tests: Monodisperse individual and clustered bio-particles are generated using an IJAG. Polydispersed biological particles are generated using puffers and the sonic nozzle. The analyses are by culturing, PCR, APS, ELISA and Coulter Multisizer. Similar to other methods, the aerosol is generated for a certain time, and the chamber air is mixed for one minute to obtain uniform aerosol concentration in the chamber. The reference filters and samplers that are being characterized sample the aerosol simultaneously for the same amount of time. At the end of sampling, the reference filters and samples are collected for analysis. Membrane filters are used to collect the bioparticles if the analysis method is by Coulter Multisizer. Samples are sent out for analyses by culturing, ELISA, and PCR.

DISCUSSION

Tests with no aerosols are conducted to determine background fluorescence of the samplers as well as reference filters. In addition, pre-washes are conducted before each test to confirm that the samplers are free of fluorescent material. After the first sampling test, up to four washes are conducted to remove the fluorescent material from the samplers, and to determine the number of washes required to remove all fluorescent material from the sampler after each test.

The use of polydisperse solid aerosols with Coulter analysis gives all particle size information at once. Therefore, it is used as a quick test to evaluate samplers. Solid particles may bounce when they hit the surfaces and reach the collection site, on the other hand, the liquid particles are removed from the air when they hit the surfaces and may not reach the collection site. After the initial quick test, if additional detailed sampling efficiency results using liquid particles are required, then the monodisperse fluorescent oleic acid particles are used with fluorometer analysis. Sampling efficiency tests using monodisperse particles are conducted one particle size at a time and it is time consuming.

Membrane filters must be used as filter medium to collect the solid particles for Coulter Multisizer and fluorometer analyses to improve sample recovery. However, the airflow rate through the membrane filters cannot be very high because of the tearing of the filters.

The use of PSL microspheres is a very convenient and accurate method for monodisperse small particles (0.5 to 3 micron) tests. They are easy to aerosolize, but are very expensive. The PSL beads have a limited amount of fluorescence, therefore very small amounts cannot be detected by the fluorometer. Also the particle count has to be significantly above the background noise for the Coulter Multisizer analysis.

The use of fluorescent oleic acid with fluorometric analysis is a very accurate method for 3 to 20 micron diameter particle tests. It is very hard to generate particles smaller than 3 microns using the VOAG because as the small orifices are used to generate smaller particles, clogging of the orifices becomes a problem. Correct excitation and emission filters should be installed in the fluorometer for the detection of sodium fluorescein. Samples have to be pH corrected for the maximum fluorescence of sodium fluorescein. All the samples have to be at the same temperature because the temperature affects the amount of fluorescence.

Much care must be taken in the use of bioaerosols in sampler characterization tests. Many aerosol generation and sampling methods kill vegetative bacteria and some spores. Another disadvantage is that some organisms start growing once they are mixed with the right nutrients, therefore, care must be taken to avoid contact with nutrient material during sampling. Samples also have to be refrigerated to prevent the growth of organisms if the analysis is planned for a later time.

CONCLUSION

Samplers are characterized at ECBC chambers at calm air conditions using the following methods: (1) monodisperse fluorescent/non-fluorescent PSL microspheres with fluorometric analysis or Coulter analysis, (2) polydisperse solid aluminum oxide particles with APS analysis or Coulter Multisizer analysis, (3) fluorescent oleic acid particles with fluorometric analysis, and (4) bioparticles with Coulter Multisizer, culturing, ELISA, PCR, and APS analyses.

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